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A STATE-OF-THE-ART LITERATURE SURVEY OF THORIA-DISPERSED NiCr FOR THE SPACE SHUTTLE THERMAL PROTECTION SYSTEMS

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March 4, 1971

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George C. Marshall Space Flight Center
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The purpose of this literature survey is to present the current status of technology for processing and fabricating thoria-dispersed nickel chrome (TD NiCr), a candidate material for the Space Shuttle thermal protection systems.

This report covers the current status of the processing and fabrication technology for TD NiCr alloy. Thoria-dispersed NiCr is a nickel-chromium base alloy strengthened by an ultrafine and highly uniform dispersion of thoria in the matrix.

Current literature indicates that TD NiCr possesses the high-temperature strength (up to 1477°K) and other desirable characteristics that make this alloy a strong candidate for Space Shuttle application.

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SUMMARY

The purpose of this literature survey is to present the current status of technology for processing and fabricating thoria-dispersed nickel chrome (TD NiCr), a candidate material for the Space Shuttle thermal protection systems.

This report covers the current status of the processing and fabrication technology for TD NiCr alloy. Thoria-dispersed NiCr is a nickel-chromium base alloy strengthened by an ultrafine and highly uniform dispersion of thoria in the matrix.

Current literature indicates that TD NiCr possesses the high-temperature strength (up to 1477°K) and other desirable characteristics that make this alloy a strong candidate for Space Shuttle application.

INTRODUCTION

General

Thoria-dispersed NiCr has been developed for long-time service in severe applications at elevated temperatures. Areas of specific application interest include thermal protection systems for high-temperature areas of the Space Shuttle. The alloy is attracting a rapidly growing interest because it possesses the following properties:

1. Outstanding oxidation and sulfidation resistance in the range 1033° to 1477°K (1400° to 2200°F).

2. Excellent strength from room temperature to 1477°K (2200°F).

3. Structural stability — insensitive to temperature excursions as high as 1589°K (2400°F).

Scope

This superalloy is now in the development stage and is produced by Fansteel, although a second source is being developed. The total amount produced last year was approximately 680 kilograms (1500 lb).

The efforts of the major aerospace companies are included in this survey; therefore, the data presented here are, to a large extent, derived from their reports.

PROPERTIES

General

1. Material Description. Thoria-dispersed NiCr is a nickel-base alloy with a composition of 20 percent chromium, 2 percent thoria, and the balance nickel. The material is strengthened by discrete, ultrafine particles of thoria uniformly distributed through the matrix. The dispersed phase is insoluble, extremely stable at elevated temperatures, and does not lower the melting point of the nickel-chromium matrix.

2. Advantages. The advantages of TD NiCr versus the disadvantages are listed below [1]:

Advantages	Disadvantages
No oxidation-protective coating	Limited availability
Good room-temperature (RT) ductility	Limited to 1477°K (2200°F) [short time to 1589°K (2400°F)]
Good high-temperature strength	Limited elevated temperature ductility
Satisfactory creep resistance	Material property data shortage
Satisfactory formability	Flatness problems (honeycomb face sheet applications)
No RT property degradation after exposure	Low welding allowables
Satisfactory fastening Mechanical Brazing	

3. Candidate Materials Comparisons. The comparative properties of leading metallic candidates are shown in Table 1 [1], wherein it may be seen that TD NiCr possesses satisfactory properties for application to 1477°K (2200°F). A summary of metallic candidates, showing the relative melting points, utilization temperature, and area of possible application, is shown in Table 2 [2]. Table 3 [2] presents the temperature limits of TD NiCr in relation to other leading candidates.

The relative unit weight of a typical panel is shown in Figure 1 [3]. Although it is heavy compared to titanium, TD NiCr has a lower unit weight than the coated refractories.

An estimated cost analysis of the candidates is shown in Figure 2 [3]; the panel cost of TD NiCr is much lower than the coated refractories.

Chemical Composition

The TD NiCr alloy furnished by Fansteel is of the following composition:

Nickel	balance
Chromium	20 wt%
Thoria	2 wt%
Carbon	less than 0.05 wt%
Sulfur	less than 0.02 wt%
Thoria particle size	less than 100 mμ or 9.6×10^{-6} cm ($> 4 \times 10^{-6}$ in.)

Thoria-dispersed NiCr was originally produced by Dupont, then sold to Fansteel. A history of the compositions is shown in Table 4 [4].

Physical Properties

Table 5 [4] lists some of the physical properties. Of particular interest is the 1667° to 1700°K (2550° to 2600°F) melting range of TD NiCr, which is higher than that of most superalloy sheet materials presently being used.

TABLE 1. PROPERTIES OF METALLIC CANDIDATES

Temperature Range	Vehicle Structural Application	Candidate Material[a]	Physical Properties[b] P S G E	Tensile Properties[b] P S G E	Creep Resistance[b,c] P S G E	Formability[b] P S G E	Weldability[b] P S G E	Oxidation Resistance[b] P S G E	Leading Candidate Materials	Remarks
1366°-1644°K (2000°-2500°F)	Lower surface leading edge and heat shield	Cr 30					Not Practical			Extremely brittle material at room temperature.
		TD Ni								Not to be used as primary structural application, not competitive with Cb alloys.
		TD NiCr 1477°K (2200°F)							*TD NiCr	Candidate uncoated material for application to 1477°K (2200°F).
		Cb[d] Alloy D-43								Poor Weldability.
		B-66								Superior density-compensated strength values, poor formability and weldability properties.
		FS-85								High density.
		C-129Y								Except for lower creep resistance, similar to Cb 752.
		Cb-752							*Cb-752	Moderately high mechanical properties.
1644°-2200°K (2500°-3500°F)		Ta[d] Alloy 90Ta-10W							*90Ta-10W	Moderate mechanical properties, very good with respect to fabricability.
		T-222								High mechanical properties, mill rolling problems.
		TZM					NA			
2200°-2978°K (3500°-4900°F)	Nose cap	W-Thoria			NA		NA		*W-Thoria	Tested in reentry profile.
		W					d			Limited by oxidation protective system.
		Zr Rod								Tested in reentry profile.

a. Maximum structural temperature limit.

b. Rating legend is as follows: E — excellent, G — good, S — satisfactory, P — poor.

c. Based on 0.5 percent creep at specified temperature.

d. With oxidation protective coating.

TABLE 2. CANDIDATE METALLICS SUMMARY

Base Metal		Melting Point, °K (°F)	Alloy Designation	Maximum Structural Utilization Temperature, °K (°F)	Area of Proposed Application
Light Metals	Aluminum	922 (1200)	2219-T81 6061-T6 7075-T6	422 (300) 394 (250)	Internal load-carrying structures, wing, tanks
	Titanium	1978 (3100)	8A1-1 Mo-1V 6A1-4V 5A1-2.5Sn	588 (600)	Internal load-carrying structures, heat shields
Superalloys	Nickel base	1728 (2650)	Inconel 718 Inco 625 René 41	1033 (1400) 1033 (1400) 1144 (1600)	External load-carrying structure — heat shields 1256°K (1800°F)
	Dispersion strengthened	1728 (2650)	TD NiCr TD Ni	1477 (2200)	External load-carrying structure — heat shields
	Cobalt base	1755 (2700)	Haynes 25 (L-605)	1255 (1800)	External load-carrying structure — heat shields
Refractory Alloy	Tantalum	3269 (5425)	90Ta-10W	1978 (3100)	Nose cap
	Columbium	2689 (4380)	Cb752	1755 (2700)	Heat shields
	Tungsten	3644 (6100)	W-2% ThO_2	2033 (3200)	Nose Cap

TABLE 3. MATERIAL TEMPERATURE LIMITS[1]

	Temperature Range, °K (°F)	
	Normal Entry	Abort
Titanium	700 (800)	755 (900)
Inconel 718	700-922 (800-1200)	755-978 (900-1300)
René 41	922-1033 (1200-1400)	978-1144 (1300-1600)
L605	1033-1256 (1400-1800)	1144-1311 (1600-1900)
TD Ni-C	1256-1478 (1800-2200)	1311-1588 (1900-2400)
Columbium C-129Y (coated)	1478-1644 (2200-2500)	1588-1756 (2400-2700)
Tantalum T-222 (coated)	1644-2200 (2500-3500)	1756-2200 (2700-3500)

1. Based on long-term technology goals [1].

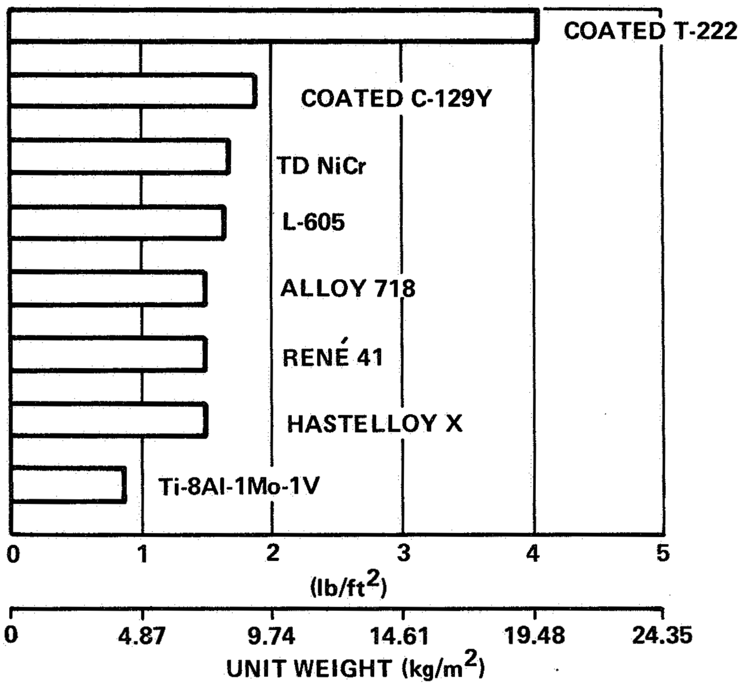

Figure 1. Thermal protection system cover panels, unit weights.

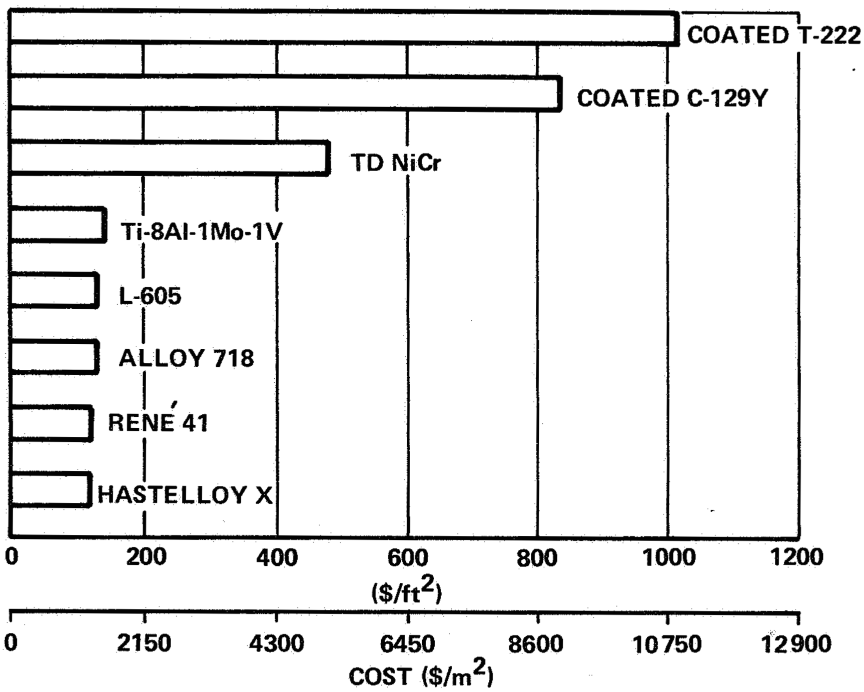

Figure 2. Thermal protection system cover panels, costs.

TABLE 4. HISTORY AND CHEMICAL ANALYSIS OF Ni-20Cr-2ThO_2, TD NiCr ALLOY

Supplier	Heat Number	Sheet Thickness, cm (in.)	N	C	S	Cr	ThO_2	Ni
Dupont	2434	0.0508 (0.020)	0.004	0.003	0.0019	20.72	2.5	Bal
Dupont	2455	0.0508 (0.020)	0.007	0.014	0.0036	21.63	2.3	Bal
Fansteel	2885	0.04572 (0.018)	0.003	0.0189	0.0036	19.34	2.0	Bal

TABLE 5. PHYSICAL PROPERTIES OF TD NiCr

Thermal Conductivity[a]		Emittance (Air)	
Temperature, °K (°F)	Conductivity $Wm^{-1}K^{-1}$ (Btu/hr/ft^2/°F/in.)	Temperature °K (°F)	E
311 (100)	14.83 (103)	589 (600)	0.08
533 (400)	17.12 (119)	700 (800)	0.15
589 (600)	18.16 (127)	811 (1000)	0.16
811 (1000)	21.27 (146)	922 (1200)	0.18
1033 (1400)	23.87 (165)	1089 (1500)	0.30
1367 (2000)	28.02 (193)	1200 (1700)	0.44
		1366 (2000)	0.57

a. Measured on TD NiCr bar
Density, 294°K (70°F) — 8.47 g/cm^3 (0.306 lb/$in.^3$)
Melting Range — 1667°-1700°K (2550°-2600°F)
Electrical Resistivity, 294°K (70°F) — 108 $\mu\Omega$-cm
Coefficient of Expansion, 294°-1256°K (70°-1800°F) —
1.584×10^{-5} m/m °K (8.8×10^{-6} in./in. °F)

Mechanical Properties

Table 6 [4] and Figures 3 and 4 [4] summarize the tensile, stress rupture, ductility, modulus, and hardness characteristics of TD NiCr sheet. Tensile strength of TD NiCr is 861.88×10^6 N/m^2 (125 000 psi) at room temperature and 11.02×10^7 N/m^2 (16 000 psi) at 1367°K (2000°F). Bend ductility, minimum bend radius through 1.83-radian (105-deg) bend, is 2T. Hardness, at room temperature, is Rc30. Figure 3 is a Larson-Miller plot for the stress-rupture properties of TD NiCr sheet. At 1367°K (2000°K) the 3.60×10^5-second (100-hr) rupture life for TD NiCr is approximately 48.26×10^6 N/m^2 (7000 psi) in the longitudinal direction and 34.47×10^6 N/m^2 to 41.37×10^6 N/m^2 (5000 to 6000 psi) in the transverse direction. Figure 4 shows the dynamic modulus of elasticity for the longitudinal and transverse directions of TD NiCr sheet, which ranges from approximately 15.17×10^{10} N/m^2 (22.0×10^6 psi) at 299°K (78°F) to 8.62×10^{10} N/m^2 (12.5×10^6 psi) at 1256°K (1800°F).

TABLE 6. MECHANICAL PROPERTIES OF TD NiCr

Temperature, °K (°F)	Tensile N/m^2 (psi)	Yield N/m^2 (psi)	Elongation (%)
Room	86.17×10^7 (125 000)	55.15×10^7 (80 000)	18
922.2 (1200)	44.81×10^7 (65 000)	37.23×10^7 (54 000)	8
1089.0 (1500)	24.13×10^7 (35 000)	20.68×10^7 (30 000)	7
1367.0 (2000)	11.02×10^7 (16 000)	10.34×10^7 (15 000)	3

Stability

The exceptionally good stability of TD NiCr sheet is indicated in Figure 5 [4], which shows 1367°K (2000°F) typical ultimate tensile strengths after exposures in various atmospheres at 1588.8°K (2400°F). Note that the strength after these exposures has decreased less than 6.89×10^6 N/m^2 (1000 psi). Stress rupture strength also remains essentially unchanged; nor is there any effect on ductility. (Figure 6 [1] gives a comparison of high-temperature materials.)

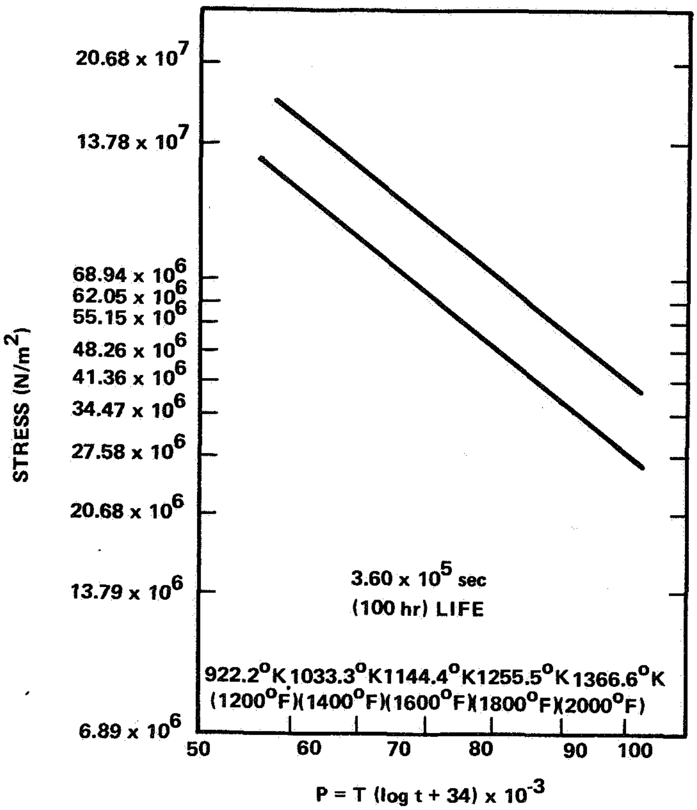

Figure 3. Larson-Miller curve for TD NiCr sheet.

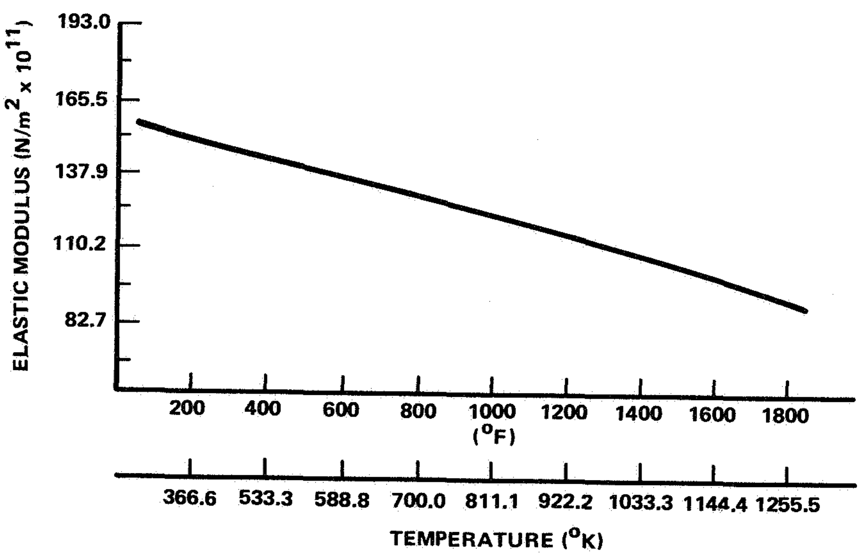

Figure 4. Dynamic modulus of elasticity of TD NiCr sheet (longitudinal and transverse directions).

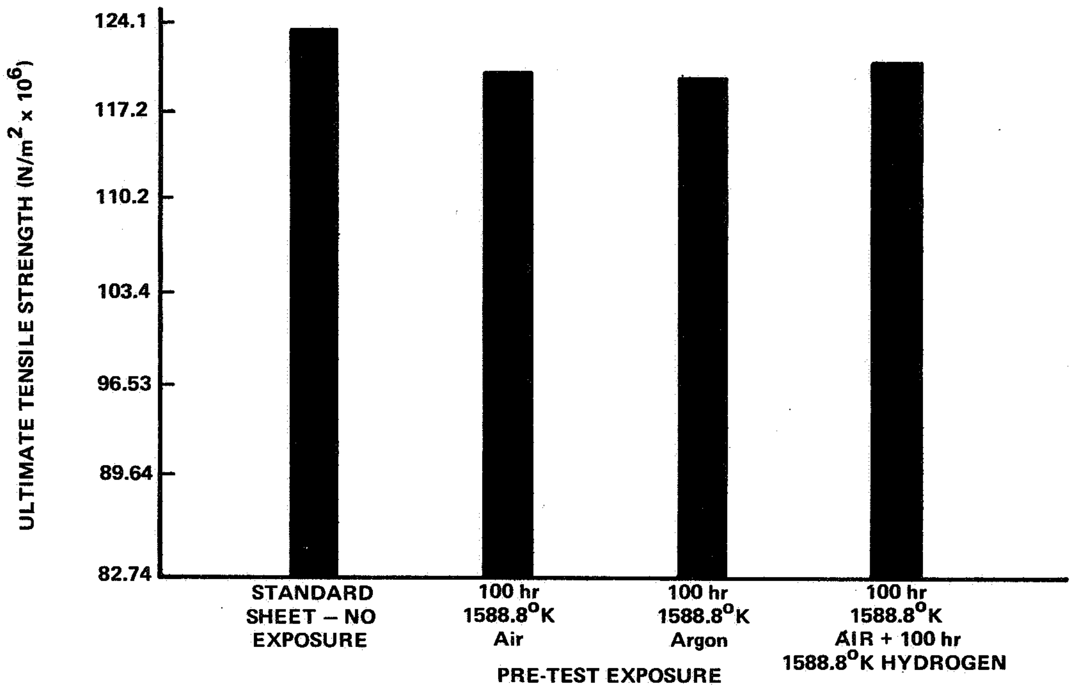

Figure 5. Effect of pre-test exposure on ultimate tensile strength at 1367°K (2000°F) TD NiCr sheet.

Oxidation Resistance

Thoria-dispersed NiCr displays outstanding resistance to static oxidation at temperatures up to 1588.8°K (2400°F). Figure 7 [4] shows the comparative cyclic oxidation behavior of TD NiCr and Hastelloy X at 1477°K (2200°F). In surface recession TD NiCr loses less than 2.54×10^{-5} meter (1 mil) per side after 1.8×10^6 seconds (500 hr) exposure in slow-moving air. Further, the oxide attack is uniform, with no detectable intergranular penetration of oxide. The slight oxide scale that forms on TD NiCr is dense, adherent, and extremely resistant to spalling under repeated thermal cycling. Figure 8 [1] compares the depth of oxidation versus temperature for the superalloys.

Current testing indicates good resistance by TD NiCr to gaseous corrosion and sulfidation effects encountered in jet engine environments and similar service applications.

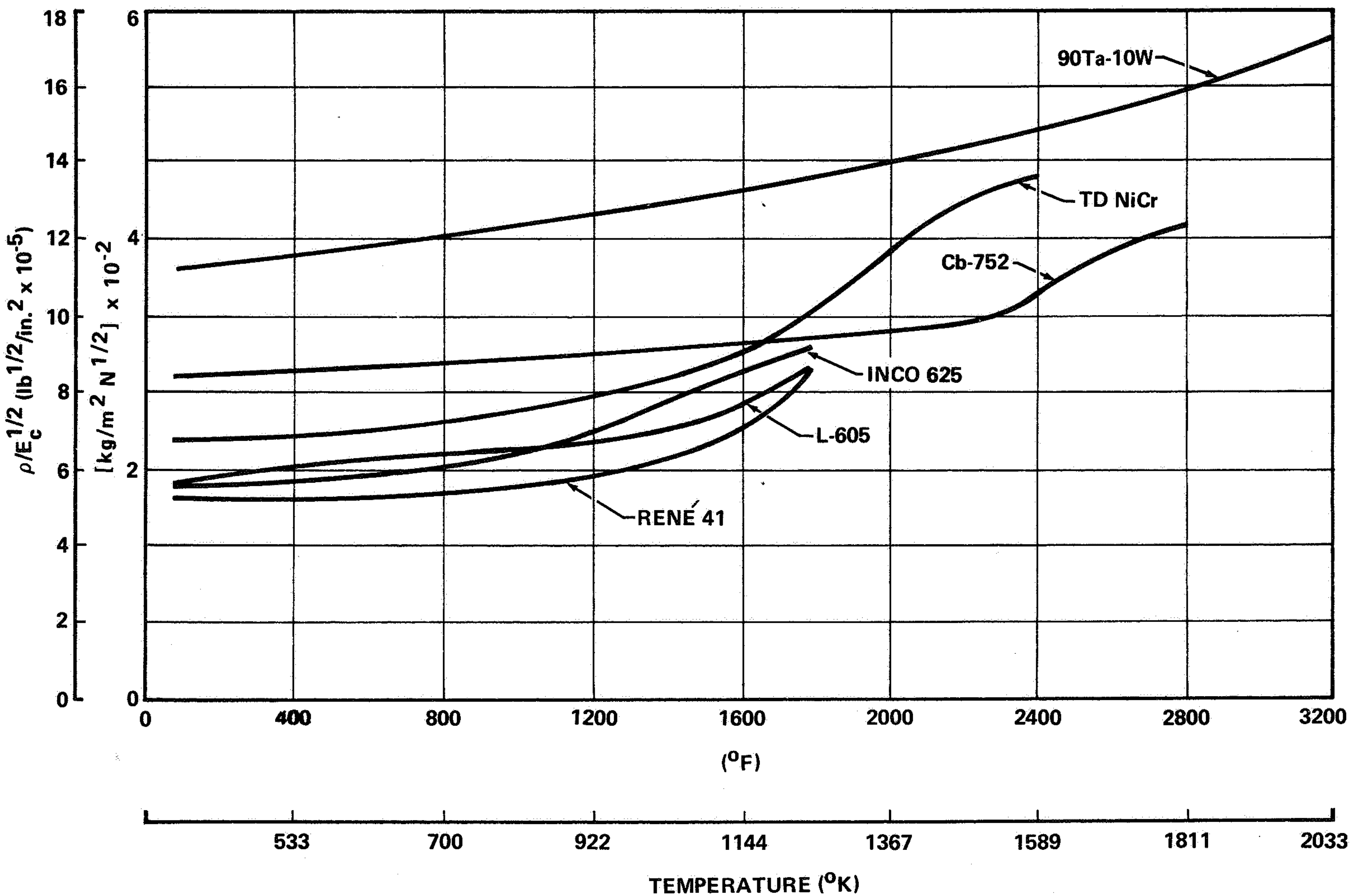

Figure 6. Structural stability comparison of candidate high-temperature materials.

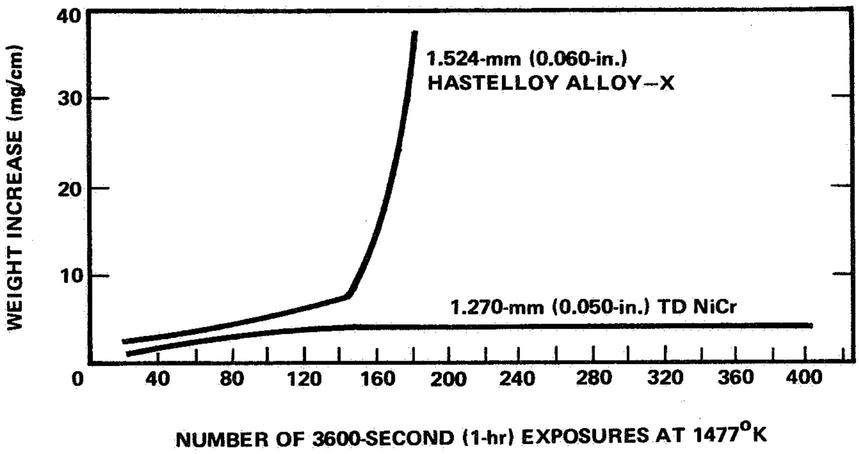

Figure 7. Cyclic oxidation of TD NiCr and Hastelloy X sheet at 1477°K (2200°F).

Thermal Fatigue Resistance

Test results indicate that TD NiCr as compared to the conventional nickel and cobalt base superalloys has excellent thermal fatigue resistance in the temperature span 1256° to 1533°K (1800° to 2300°F).

Thoria

1. Radioactivity and Radiation. Radioactivity is the spontaneous emission of particles and rays by the disintegration of nuclei of atoms.

The radioactivity of thoria is a property of the element thorium. The particles and rays referred to have been classified as three distinct types: alpha, beta, and gamma, designated by the Greek letters.

Alpha particles are relatively massive, and while they are emitted at great speed they travel no more than a few centimeters in air. Several layers of paper are a sufficient shield to stop them. Beta particles are electrons and penetrate farther; several meters is the approximate distance in air. They can be stopped by a thin sheet of aluminum. Gamma rays are the most penetrating; they are similar to X-rays but more energetic.

The energy emitted by a radioactive substance is termed radiation, and it can be measured easily. A Geiger counter (survey meter) measures direct gamma and beta radiation; a proportional counter measures direct alpha radiation. In addition, film badges or dosimeters are used to record cumulative exposure to beta and gamma radiation by an individual.

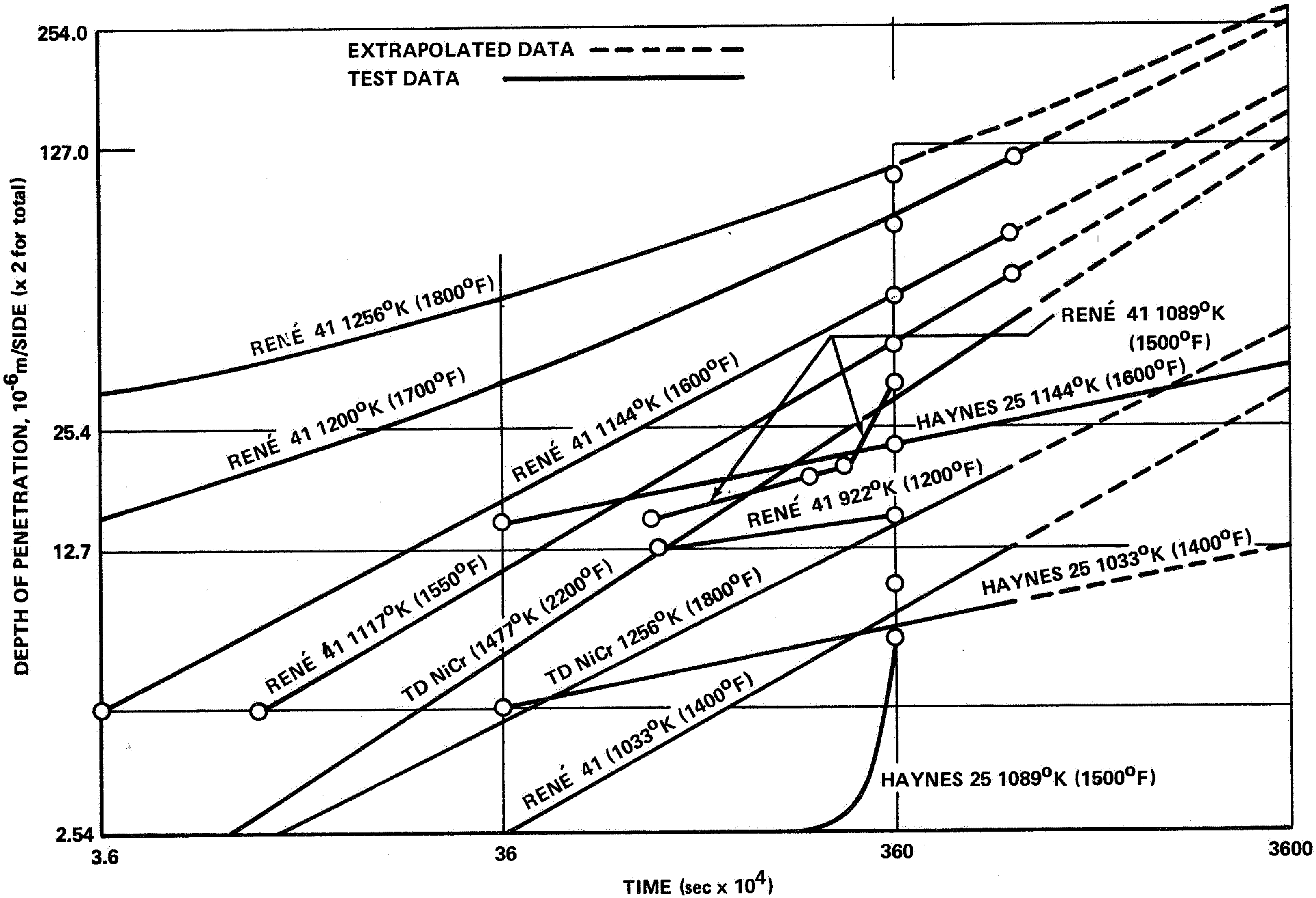

Figure 8. Depth of oxidation versus temperature for René 41, TD NiCr, and Haynes 25.

Gamma radiation is measured in roentgens and alpha radiation in disintegrations per unit time, both of which can subsequently be converted to rads, the unit of measurement of total exposure.

2. Thoria Dispersion. When metals are subjected to stress at elevated temperatures, their creep strength, or ability to sustain applied loads without deformation and ultimate rupture, progressively decreases as temperature increases. As a consequence, when designing for high-temperature performance the engineer is confronted with reduced strength and lower operational efficiency. The dispersion of thoria in metals and alloys by the Fansteel process promotes a marked increase in high-temperature creep strength and in high-temperature tensile strength.

Exposure to radiation can arise from proximity to a radioactive source and from such amounts of radioactive material as are introduced into body tissue through cuts and abrasions or into the lungs and the blood stream by inhalation of dust and fumes.

Exposure to radiation, as such, either by proximity, ingestion, or inhalation is not, in itself, a difficult problem in the handling of nickel-thoria metal dispersions. Permissible amounts or "doses" of radiation have been established by the Atomic Energy Commission (AEC). These allowable doses are expressed in amounts per calendar quarter or per 40-hour week. While allowable dose varies as to the age, intervals and duration of exposure, and past radiation history of an individuals, the radiation associated with materials containing 2 percent thoria is appreciably below the currently established allowable limits.

An indication of this is shown in Tables 7 and 8 [5]. Table 7 presents the results of air counts made during various fabrication operations; Table 8 shows the results of measurements made at varying distances from nickel-2 percent thoria bar and sheet, unpackaged and packaged.

3. AEC License Requirements. Thorium is designated as a "source" material by AEC. A source material is one which contains by weight more than one-twentieth of one percent of uranium or thorium or any combination of these. Thoria is a combination of thorium and oxygen. The amount of thoria added to materials in the Fansteel process will vary from 1 to 4 percent, depending on the properties desired. Accordingly, these metals are source materials and as such come under the license requirements of the AEC.

TABLE 7. AIRBORNE ALPHA ACTIVITY[1] FROM FABRICATING OPERATIONS

Operation[a]	Concentration	
Abrasive sanding (wet)	0.009×10^{-13}	AEC maximum permissible level of radiation for restricted area for any seven days is 3.00×10^{-11} μc/ml.
Hot forging	0.007×10^{-11}	
Sand blasting	0.020×10^{-11}	
Lathe turning	0.023×10^{-11}	
Surface grinding (dry)	0.030×10^{-11}	
Welding (14.24 cm from source)	0.171×10^{-11}	

a. With the exception of welding, all measurements at breathing zone level approximately 61 cm from source.

TABLE 8. EXTERNAL RADIATION MEASUREMENTS — BAR AND SHEET STOCK — BETA AND GAMMA EMISSIONS ($mr/3.6 \times 10^3$ sec)[2]

Distance mm (in.)	19.4-kg (43-lb) 1.52×10^{-3}-m (0.060-in.) Sheet		14.4-kg (32-lb) 57.2×10^{-3}-m (2.25-in.) dia Bar	
	Unpackaged	Packaged[a]	Unpackaged	Packaged[a]
Surface	0.30	0.15	0.43	0.30
25.4 (1)	0.27	0.14	0.30	0.22
152.4 (6)	0.15	0.09	0.13	0.09
304.8 (12)	0.07	0.06	0.07	0.04
609.6 (24)	nil	nil	0.03	0.02

Note: AEC limits for external radiation in a restricted area permit an average of $2.5\ mr/3.6 \times 10^3$ sec (2.5 mr/hr).

a. Package for sheet: wood (12.7 mm thick) crating.
Package for bar: cardboard tube 12.7-mm wall.

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1. N.M.C. Model PC-3A proportional counter.

2. Tracer Lab survey meter Model SU14.

Licenses are of two types: general and specific. General licenses are considered to have been issued to any person who does not use or transfer more than 6.8 kilograms (15 lb) of contained source material (e.g., thorium) at any one time or a total of more than 68.04 kilograms (150 lb) in a calendar year. For a 2 percent thoria dispersion (e.g., TD nickel) this would represent a total of 340.2 kilograms (750 lb) at any one time or 3402 kilograms (7500 lb) a year. A 4 percent thoria dispersion would represent an allowable total for general license of 170.1 kilograms (375 lb) at one time and 1700 kilograms (3750 lb) for a year. General licenses do not have to be applied for, and persons operating under them are exempt, for the most part, from the AEC regulations (Title 10 CFR, Part 20) relating to protection against radiation.

If the use and transfer involves quantities greater than those indicated above, a specific license is required. Application is made to: Licensing Control Branch, U.S. Atomic Energy Commission, Washington, D. C. 20545.

The general procedures specified by the AEC with respect to all matters relating to use, allowable dosage, environmental concentrations, safety procedures, and the like are clearly delineated in Title 10 CFR, Parts 20 and 40. The requirements do not discourage the industrial or commercial use of radioactive materials, providing that public health and safety are properly protected.

It is not difficult to obtain a specific license. Fansteel is prepared to offer guidance in the necessary procedures [5]. Equipment required for radiation measurement and personnel monitoring is available from numerous scientific equipment sources and is selected with respect to the specific fabricating situations.

4. Why Use Thoria? The fact that thoria is radioactive, to a relatively minor degree, has nothing whatsoever to do with the favorable properties which result from its dispersion in many materials. The benefits derive principally from the fact that thoria is very stable at high temperatures. It is a refractory oxide, which is to say it can withstand great heat without physical change.

When finely dispersed in a material, the thoria inhibits the movement of the matrix atoms under stress and as a result strengthens the material at elevated temperatures. Unlike other methods, the production technique effects the dispersion of thoria by chemical rather than mechanical means.

Availability

Thoria-dispersed NiCr sheet is presently being produced in developmental quantities in the following sizes:

Gage mm (in.)	Width mm (in.)	Length mm (in.)	Weight kg/m^2 (lb/ft^2)
0.508 (0.020)	609.6 (24)	up to 1219.2 (48)	4.29 (0.88)
0.762 (0.030)	609.6 (24)	up to 914.4 (36)	6.44 (1.32)
1.016 (0.040)	609.6 (24)	up to 914.4 (36)	8.64 (1.77)
1.270 (0.050)	609.6 (24)	up to 914.4 (36)	10.74 (2.20)
1.524 (0.060)	609.6 (24)	up to 914.4 (36)	12.74 (2.63)
1.905 (0.075)	609.6 (24)	up to 762 (30)	16.11 (3.30)

Thoria-dispersed NiCr metal is availabe in sheet and rod. Other forms are also available on a development basis from Fansteel. Until recently Fansteel has been the only source of this alloy. However, Sherrit-Gordon Mines Limited is in the process of becoming a second source. The total output by Fansteel this current year is approximately 680 kilograms (1500 lb), sponsored by NASA.

Safety Precautions

The radiation associated with materials containing 2 percent thoria is appreciably below the currently established allowable limits, where quantities handled do not exceed 340 kilograms (750 lb) at one time or 3402 kilograms (7500 lb) a year. However, if the use of TD NiCr exceeds this amount it is recommended that the full aspect of the safety requirement be investigated and a safety procedure established.

FABRICATION

General

Thoria-dispersed NiCr is supplied in the stress-relieved condition and will have a Rockwell B hardness ranging from 85 to 100 for bar and from 80 to 85 for sheet. Mechanical properties depend on the cold work-stress relief cycle and may be affected if the material is subjected to severe reductions without intermediate anneals.

Forming

1. General. Limited experimental tests indicate that many forming methods may be applied to TD NiCr sheet. Hemispherical shapes have been made by spinning and hydroforming. Brake formed parts have also been produced.

Cup testing provides some measure of formability. Laboratory cup tests were made, and cup depth at fracture ranged from 7.62 to 10.16 millimeters (0.3 to 0.4 in.) in 0.762- to 1.27-millimeter (0.030- to 0.050-in.) sheet. This test employed a restrained edge, 19.05-millimeter (0.75-in.) round punch and 31.75-millimeter (1.25-in.) die.

Preliminary work accomplished by Convair and others demonstrates that TD NiCr can be formed at room temperature by conventional forming methods. The effect of various amounts of cold working (prestrain) on the mechanical properties of TD NiCr sheet supplied in the stress-relieved 1367°K (2000°F) condition has also been determined in preliminary tests. However, continued work should be performed to determine the prestrain limits and the combined effects of inter-stage heat treatments and subsequent cold work on the mechanical properties, particularly for thin-gage sheet [6].

2. Material Conditions. The mill-supplied conditions for all TD NiCr sheet has been in the stress-relieved condition after rolling, with the mill stress-relief treatment performed at 1561.1°K (2350°F). Although room-temperature ductility is good, the material exhibits poor high-temperature ductility, thus resisting the use of elevated temperature forming.

Preliminary information from Fansteel indicates that TD NiCr, in the as-rolled (non-recrystallized) condition and without a subsequent stress relief heat treatment, has exceptional elongation at elevated temperature [35 percent at 922°K (1200°F)]. This is in contrast to the elongation of the normally supplied stress-relieved material with no better than 20 percent elongation at room temperature and 12 percent at 922°K (1200°F).

3. Male and Female Die Forming. A development program in forming beaded heat shield panels has been recently completed by Convair [7].

Beaded heat-shield panels for radiative thermal protective systems were formed with hydropress equipment and the Verson-Wheelon press, which employs hydraulic fluid for the pressure medium. Female die forming was necessary to avoid panel buckling in the initial forming stages. Additional forming stages continued in male dies. Best results were attained by forming

TD NiCr materials in combination with a caul sheet of 0.305-millimeter- (0.012-in.) -thick type 321 stainless steel sheet. The workpiece was located between the caul sheet and forming force. A "picture frame" type of pressure plate over the panel border with the opening close to the bead configurations is a key item in the prevention of forming buckles.

Two types of stage forming were applied to the TD NiCr beaded panels. First, stage forming was done in the female die (Fig. 9), after which the material was stress relieved 5.4×10^3 seconds (1.5 hr) at 1561°K (2350° F). Additional forming stages were continued in the male die (Fig. 10) [8] until 8 to 10 percent total stretch was reached. At this point the material was again stress relieved 5.4×10^3 seconds (1.5 hr) at 1561°K (2350° F). Pressures in the range of 25.86 to 28.96×10^6 N/m^2 (3750 to 4200 psi) were required in the male and female die forming operations. A summary of the beaded heat shield forming operations is presented in Table 9 [2].

4. Influence of Work Hardening on the Uniform Percent Elongation of TD NiCr Material [8]. Materials with work hardening properties are characterized by uniform elongation when strain specimens are pulled in the plastic region, the degree of which depends on the crystal structure, specimen geometry, strain rate, and many other conditions. Uniform elongation changes to nonuniform elongation with increased magnitude in a delta length, l, of the specimen under continued loading in the plastic region. The change to nonuniform elongation is a signal of impending specimen failure; thickness and width decrease, and these decreases localize at some point to precipitate a reduction of area associated with the fracture mode. During this occurrence in TD NiCr material, another delta length, l_1, of the strain specimen retained uniform deformation after the specimen fractured elsewhere. It is a measure beyond which forming deformations are subject to fracture.

5. Actual Formability Limits of TD NiCr Material [8]. Studies by Convair in 1967 demonstrated work hardening characteristics of TD NiCr material from an examination of tensile strain specimens. The specimens had a reduced section 25.4 millimeters (1 in.) in width in a 254-millimeter (10-in.) gage length. Material thickness was 1.09 millimeters (0.043 in.). The percent elongation did not deviate more than ±1.5 percent from 10.4 percent total stretch. The maximum uniform percent stretch within ±1.5 percent is, therefore, in the range of 10 to 11 percent and was attained from as-received material with 17.5 percent elongation.

The limit of 10 to 11 percent uniform elongation in tensile stretching was approached by stretch-wrap forming of TD NiCr angles with

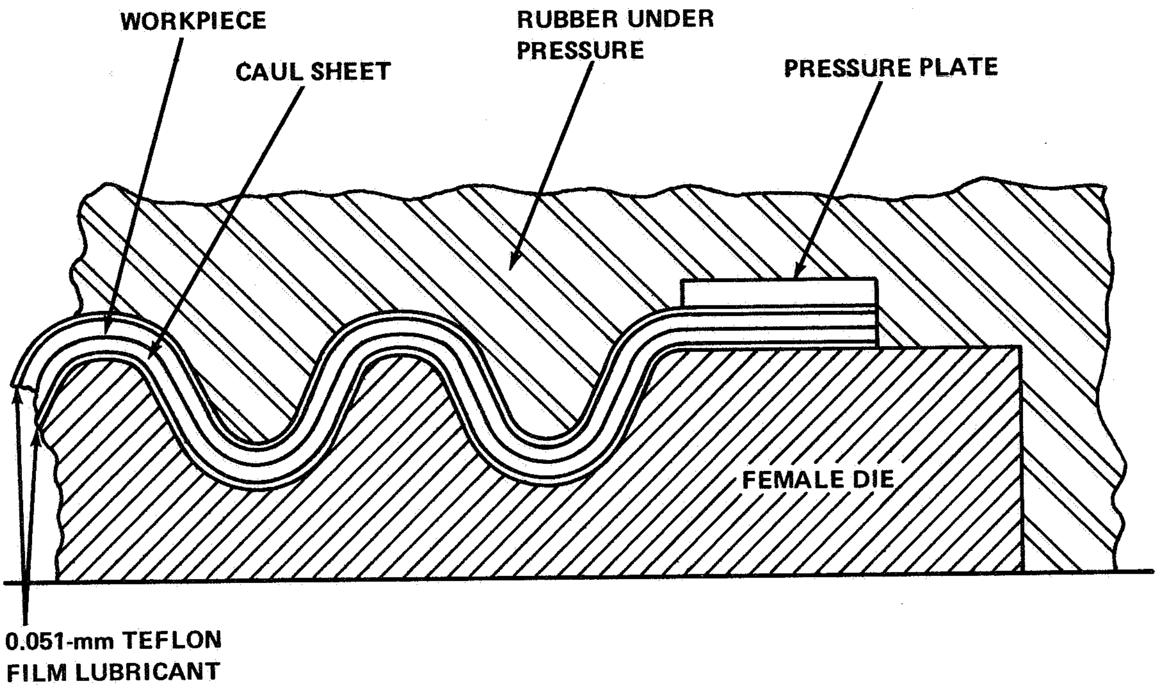

Figure 9. Lay-up of workpiece in a female die.

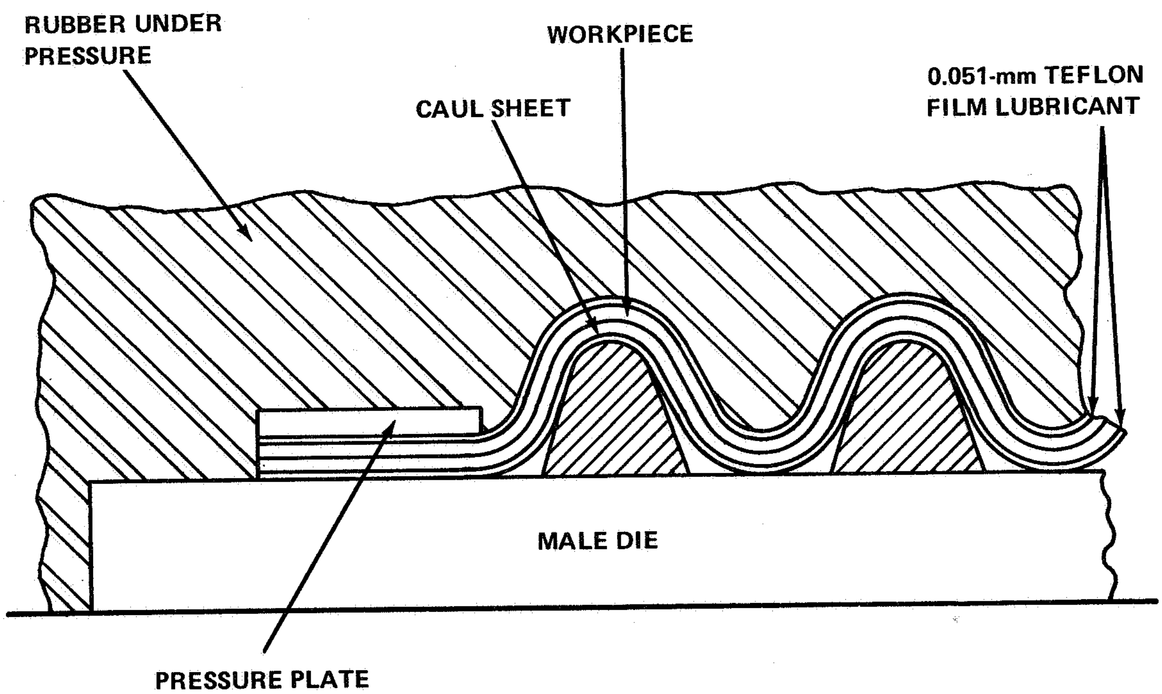

Figure 10. Lay-up of workpiece in a male die.

TABLE 9. SUMMARY OF BEADED PANEL FORMING EVALUATION WITH TD NiCr ALLOY SHEET

Panel No.	Material	Forming Equipment	Caul Sheet	Bead Height, mm (in.)		Bead Stretch, %[a]		End Border Neck-In, mm (in.)	Remarks
				End Beads	Center Beads	End Beads	Center Beads		
				Female Die Forming					
III	Dupont, 0.275 mm (0.011 in.) Thick	Verson-Wheelon $27.58 \times 10^6\ \mathrm{N/m^2}$ (4000 psig)	Yes	5.08 (0.20) L.H. 5.84 (0.23) R.H.	5.59 (0.22)	2.0	4.5	3.30 (0.13)	Passed. Stress relieved after first female forming operation.
III	Dupont, 0.301 mm (0.012 in.) Thick	Verson-Wheelon $25.86 \times 10^6\ \mathrm{N/m^2}$ (3750 psig)	Yes	7.11 (0.28)	5.84 (0.23)	5.0	6.5	—	Pased after second female forming operation.
IV	Dupont, 0.301 mm (0.012 in.) Thick	Verson-Wheelon $27.58 \times 10^6\ \mathrm{N/m^2}$ (4000 psig)	Yes	5.33 (0.21) L.H. 5.84 (0.23) R.H.	5.84 (0.23)	3.0	4.5	3.30(0.13)L.H. 4.57(0.18)R.H.	Passed. Stress relieved after first female operation.
IV	Dupont, 0.301 mm (0.012 in.) Thick	Verson-Wheelon $25.86 \times 10^6\ \mathrm{N/m^2}$ (3750 psig)	Yes	7.37 (0.29)	6.35 (0.25)	6.0	6.0	—	Passed after second female forming operation
VII	Fansteel, 0.301 mm (0.012 in.) Thick	Verson-Wheelon $28.96 \times 10^6\ \mathrm{N/m^2}$ (4200 psig)	Yes	5.59 (0.22)	5.59 (0.22)	3.0	6.0	3.81 (0.15)	Passed. Stress relieved after this forming operation.
				Male Die Forming After Female Die Forming					
III	Dupont, 0.275 mm (0.011 in.) Thick	Verson-Wheelon $27.58 \times 10^6\ \mathrm{N/m^2}$ (4000 psig)	Yes	7.62 (0.30) L.H. 7.87 (0.31) R.H.	7.62 (0.30)	5.0	8.5	—	Passed. Stress relieved after first male forming operation.
III	Dupont, 0.275 mm (0.011 in.) Thick	Verson-Wheelon $28.96 \times 10^6\ \mathrm{N/m^2}$ (4200 psig)	Yes	7.62 (0.30)	7.62 (0.30)	6.0	10.5	—	Passed after second male forming operation. Increased stretch due to improved bead definition.
III	Dupont, 0.275 mm (0.011 in.) Thick	Verson-Wheelon $24.13 \times 10^6\ \mathrm{N/m^2}$ (3500 psig)	Yes	8.13 (0.32)	7.62 (0.30)	6.5	12.0	4.57 (0.18)	Passed after sizing operation.
IV	Dupont, 0.301 mm (0.012 in.) Thick	Verson-Wheelon $27.58 \times 10^6\ \mathrm{N/m^2}$ (4000 psig)	Yes	7.62 (0.30)	7.62 (0.30)	—	—	—	Passed. Stress relieved after first male forming operation. Stretch from this operation not recorded.
IV	Dupont, 0.301 mm (0.012 in.) Thick	Verson-Wheelon $24.13 \times 10^6\ \mathrm{N/m^2}$ (3500 psig)	Yes	7.62 (0.30)	7.62 (0.30)	2.0	2.5	—	Passed. The percent stretch is due to sizing to contour and is not total stretch from all forming operations.
VII	Fansteel, 0.301 mm (0.012 in.) Thick	Verson-Wheelon $28.96 \times 10^6\ \mathrm{N/m^2}$ (4200 psig)	Yes	7.11 (0.28)	6.60 (0.26)	5.0	10.0	—	Passed. Stress relieved after first male forming operation.
VII	Fansteel, 0.301 mm (0.012 in.) Thick	Verson-Wheelon $28.96 \times 10^6\ \mathrm{N/m^2}$ (4200 psig)	Yes	7.62 (0.30)	7.62 (0.30)	5.0	12.5	—	Passed after second male forming operation.
VII	Fansteel, 0.301 mm (0.012 in.) Thick	Verson-Wheelon $28.96 \times 10^6\ \mathrm{N/m^2}$ (4200 psig)	Yes	8.13 (0.32)	7.87 (0.31)	6.0	13.5	5.08 (0.20)	Passed after sizing operation.

a. The percent stretch, except as noted for Panel IV, includes deformations from all prior forming operations.

0.864-millimeter (0.034-in.) material thickness. Maximum stretch was in the range of 8 to 9.5 percent and material thickness decreases 0.0375 millimeter (0.0015 in.) for 9.5 percent maximum stretch.

Stress relieving between strain applications is beneficial to increase the available ductility between forming stages. Stress relieving as-received 1.092-millimeter (0.43-in.) -thick TD NiCr material for 7.2×10^3 seconds (2 hr) at 1367°K (2000°F), after 10 percent prestrain, increases the percent elongation to 24 percent in 25.40 millimeters (1 in.), up from 17.5 percent in 25.40 millimeters (1 in.). These findings point up the need to conduct an in-depth study of additional stress-relieving treatments after various amounts of strain application to determine the magnitude of maximum formability from increased ductility.

6. Forming Z-Sections and Edgemembers. The Hypersonic Aerospace Vehicle Structures Program, recently completed by Martin Marietta Corporation [9], resulted in the following recommended procedures.

Shear blank stock for forming

Deburr and straighten

Stress relieve nonflat sections

Brake form, rubber mold form and coin. Match thickness and Z-leg height.

Rough size leg height and flange and stress relieve

Resize

Stress relieve

Final size and dimension.

It was determined that the intermediate stress-relief operations stabilized the parts suitable for assembly.

Machining

Thoria-dispersed NiCr can be machined by techniques used for iron-base alloys. However certain requirements are imposed by the high strength of the nickel alloys, their tendency to work harden, and their "gumminess" in some conditions [10].

1. Work Hardening. Nickel alloys, like austenitic stainless steels, work harden rapidly. The high pressures developed during machining produce a hardening effect that retards further machining and may also cause distortion in parts that have small cross sections.

There are two methods of reducing work hardening during machining. One is to work harden the material before machining by cold deformation. The second method is to employ careful machining practices. Sharp tools with positive rake angles, which cut the metal instead of pushing it, are required. Feed rate and depth of cut must be sufficient to prevent burnishing or glazing. Tools should not be allowed to rub the work, either by improper clearance or by being allowed to dwell in the cut.

2. Distortion. Even with the best machining conditions, some stresses are produced that may cause subsequent distortion of the work. For maximum dimensional stability, it is best to rough out the part almost to size, stress relieve it, and then finish it to size. Stress relieving has little effect on dimensions but may affect mechanical properties.

3. Microstructure. Grain size has little direct effect on the machinability of nickel alloys. In general, microstructure affects machinability in two ways:

a. The presence of graphite or sulfide phases greatly improves machinability.

b. Hard phases, such as carbides, nitrides, carbonitrides, oxides, silicates, and possibly also the gamma-prime phase Ni_3 (Al, Ti), are abrasive and cause rapid tool wear.

The NiCr alloys are less abrasive than the common grades of austenitic stainless steel because they have lower carbon content and therefore fewer carbide particles.

4. Cutting Fluids. Almost any cutting fluid, or none, can be used in machining nickel alloys. In many applications, nickel alloys respond well to ordinary sulfurized mineral oil; sulfur imparts improved lubricity and antiweld properties. If the temperature of the oil and workpiece becomes high enough during machining to cause brown sulfur staining of the work, the stain

can be readily removed with a cleaning solution of the sodium cyanide or chromic-sulfuric acid type. This should be done before any thermal treatment, including welding, because during further exposure to high temperature, the staining may cause intergranular surface attack. To avoid intergranular corrosion, the parts should be immersed in cleaning solution only long enough to remove the stain. High-speed machining operations that create high temperatures might preclude the use of a sulfurized oil because of sulfur embrittlement of carbide tools. (Many sintered carbides have a nickel or cobalt matrix that is sensitive to sulfur attack at high temperature.) However, flooding the cutting area with cutting fluid generally cools the tool enough to avoid breakdown of the carbide bond.

Water-base fluids are preferred in high-speed turning, milling, and grinding because of their greater cooling effect. These may be soluble oils or chemical solutions. Except for grinding, which depends almost entirely on cooling and flushing, some chemical activity is always desired, and is generally provided by chlorine, amines, or other chemicals.

For slower operations, such as drilling, boring, tapping, and broaching, heavy lubricants and very rich mixtures of chemical solutions are needed. Oils should be used when drilling Nickel 200 and Inconel X-750. In the drilling and tapping of small-diameter holes and in other operations in which lubricant flow and chip flushing are restricted, chlorinated hydrocarbon solvents such as trichlorethylene and trichloroethane will improve performance. These less viscous fluids can be used alone or can be used for diluting mineral and lard oils.

A cutting fluid of the spray-mist type serves adequately for simple turning operations on TD NiCr [10].

5. Turning. Single-point turning tools used for cutting nickel alloys should have positive rake angles so that the metal is cut instead of pushed, which would occur if negative rake angles were used. A secondary function of the rake angle is to guide the chip away from the finished surface. The side cutting-edge angle is second in importance only to the rake angle.

The nose radius, which joins the end and side cutting edges, strengthens the tool nose and helps to dissipate the heat generated in cutting.

6. Drilling. In drilling nickel alloys, steady feed rates should be used. If the drill is allowed to dwell, excessive work hardening of the metal at the bottom of the hole will make it difficult to resume cutting and may

result in breaking of the drill when it does take hold. The setup should be as rigid as possible. Stub drills are recommended. Drill jigs should be used whenever possible. The drills have a 2.06-radian (118-deg) point angle, a helix of about 0.52 radian (30 deg), a 0.20-radian (12-deg) lip relief angle, and a chisel-edge angle of 2.18 to 2.36 radians (125 to 135 deg).

Heavy-duty, high-speed steel drills with a heavy web are recommended for drilling TD NiCr. Cobalt-bearing high speed steel drills give longer tool life. Cutting pressures are reduced and a positive effective rake maintained if the web is thinned at the chisel point. Increasing the point angle to 2.36 radians (135 deg) is helpful.

7. Reaming. Fluted reamers for nickel alloys are produced as standard items and are characterized by

High-speed steel tool material

Right-hand cut

Right-hand helix (positive axial rake)

Positive radial rake.

Operating speed for reaming should be about two thirds the speed for drilling the same material, but not so high as to cause chatter. Other factors contributing to chatter are lack of rigidity in the setup, misalignment, and dull tools.

Reamer feed into the work should be 0.0381 to 0.102 millimeter (0.0015 to 0.004 in.) per flute per revolution. Too low a feed rate will result in glazing of the work and excessive wear of the tool. An excessive feed rate reduces the accuracy of hole dimensions and the quality of the finish. In reaming nickel alloys, sufficient stock must be removed so that non-work-hardened or nonglazed material is being cut. Good starting points for stock removal are 0.254 millimeter (0.010 in.) for a 6.35-millimeter (0.25-in.) hole, 0.375 millimeter (0.015 in.) for a 12.7-millimeter (0.5-in.) hole, and up to 0.635 millimeter (0.025 in.) for a 38.1-millimeter (1.5-in.) hole.

Reamers must be kept sharp; honed reamers produce smoother surfaces and last longer between grinds.

8. Milling. The essential requirements of milling are accuracy and smooth finish, and therefore it is imperative to have sharp tools and rigid machines and fixtures. High-speed steel cutters (M2 and M10) are most suitable, particularly for interrupted cutting action.

Chip problems in milling are the same as in turning. Standard milling cutters provide adequate clearance for chips.

Heavy-duty milling cutters with 0.21-radian (12-deg) positive radial rake and 0.785-radian (45-deg) axial rake are preferred for rough milling all alloys except those of group D-2. Light-duty cutters with 0.21-radian (12-deg) positive radial rake and 0.31-radian (18-deg) axial rake (helical flutes) are best for the high-strength alloys of this group. They require low cutting speeds 0.06 to 0.10 Sm/sec (10 to 20 sfm) and light chip loads. The light-duty cutters have more teeth than the heavy-duty type; consequently, light-duty cutters operate at higher cutting rates for the cutting speeds allowed.

Finishing cutters for all alloys should be of the high-helix type with 0.26-radian (15-deg) positive radial rake and 0.807- to 1.13-radian (52- to 65-deg) helical flutes (positive axial rake). Staggered-tooth cutters, with alternate teeth of opposite helix, are best for milling grooves. High-speed steel slitting saws with side chip clearance are recommended for narrow slotting.

9. Band Sawing. Band sawing can be used for cutting off all nickel alloys. High-speed steel saws with flexible backs are recommended. Raker-set teeth are suggested for sawing all forms of material other than light-gage sheet and thin-walled tube. Saws with wavy-set teeth are best for sawing thin sections.

10. Abrasive Cutoff. All nickel alloys can be cut off with abrasive wheels. For dry cutting small sections [up to 25.4 millimeters (1 in.)], aluminum oxide resinoid-bond wheels are satisfactory.

Wet cutting is preferred for sections over 25.4 millimeters (1 in.) thick. Aluminum oxide rubber-bond wheels such as A-602-M-R are recommended. Water with a rust inhibitor is a satisfactory grinding fluid. Speed should be about 25.4 to 27.9 Sm/sec (5000 to 5500 sfm) feed, the maximum permitted by machine capability.

11. Grinding. Methods of grinding nickel alloys do not differ greatly from those for steel. For best results, nickel alloys should be ground wet.

A solution of 0.094 m^3 (25 gal) of water and 0.45 kilogram (1 lb) of sal soda or 50 parts of water to 1 part of soluble oil is a suitable grinding fluid for operations other than crush and thread grinding. Grinding oil is the best fluid for crush and thread grinding. Sodium chromate may be added to the sal soda solution to inhibit rusting of the machine and circulation system.

PROCESSING

Heat Treatment

1. General. Thoria-dispersed NiCr is not age-hardenable by heat treatment. The thoria is inert with respect to the nickel-chromium matrix, hence solution and aging reactions do not occur. The alloy does work-harden during fabrication. Room temperature ductility can be recovered by heat treating at 1367°K (2000°F) for 3.6×10^3 to 7.2×10^3 seconds (1 to 2 hr) in hydrogen or cracked ammonia.

2. Stress Relieving. A program was recently completed by Martin Marietta Corporation in forming Z-sections, doublers, and edgemembers for TD NiCr brazed panels [11].

To establish a stress-relieving temperature, a series of TD NiCr sheet specimens of varying degrees of cold work was stress-relieved at 1506°, 1589°, and 1617°K (2250°, 2400°, and 2450°F) for 3600 seconds (1 hr).

On the basis of tensile strength results and ductility, the 1561°K (2350°F), 3600-second (1-hr) stress-relieving straightening cycle is applicable to both doubler and Z-frame parts. Removing stock distortion is accomplished by loading the surface of the doubler with $12.7 \times 25.4 \times 101.6$-millimeter ($0.5 \times 1 \times 4$-in.) tungsten blocks, and in the case of Z-sections, tungsten pellet load is distributed over the Z-frame long flange surface and the tungsten blocks on the sheet flange. A precision sized spacer for the leg height is used to size-straighten the member.

The results of this program indicate that the stress-relieving temperature for formed parts is 1561°K (2350°F) for 3600 seconds (1 hr). A stress-relief fixture is also desirable to prevent distortion and also provide a straightening process to remove bowed conditions, oil canning, etc.

Intermediate stress-relieving operations were also found to be necessary during multiple forming operations.

Descaling and Pickling

Light oxide scales can be removed by acid pickling. Recommended solutions are:

	Solution No. 1	Solution No. 2
Water, m^3 (gal)	0.011 (3)	0.004 (1)
Nitric Acid 96.52-cm (38-in.) Baume, m^3 (gal)	0.004 (1)	0.004 (1)
Hydrofluoric 40%, m^3 (gal)	0.757×10^{-3} (1/5)	0.473×10^{-3}
Temperature, °K (°F)	322-333 (120-140)	294-311 (70-100)

Removal of heavy scale can be accomplished in fused caustic such as Du Pont sodium hydride (Du Pont Electrochemicals Department) or "Virgo" descaling bath (Hooker Chemical Company).

The requirements for a cleaning procedure for chemically etched TD NiCr with a rough surface are noteworthy. The normal cleaning required on a standard ground finish for this alloy is merely a light degreasing with acetone or similar solvent. With the chemically etched rough surface, there is sufficient contamination to inhibit brazing to some extent. A procedure using hot detergent cleaning in an ultrasonic bath has been found to be the most effective method of removing the etching contaminants [11].

Cleaning

Cleaning of TD NiCr prior to joining by welding, brazing, or bonding can be divided generally into degreasing and the removal of light and heavy oxide scales. An alternate to scale removal is a means for preventing the formation of such scales during thermal treatments.

Degreasing of the material is readily accomplished by solvent vapor degreasing or by wiping. Ultrasonic cleaning in hot detergent solutions is effective for removing contaminants from chemical cleaning or chemical milling operations prior to brazing.

The removal of the highly tenacious light and heavy oxide scales requires special techniques, which are in need of further investigation. Chemical cleaning solutions of nitric-hydrofluoric acids at 344°K (160°F) did a modest job of light scale and oxide removal. However, the complete removal of moderate to heavy scales using hot acid solutions was not completely successful, indicating the need for more investigation. The removal of heavy scales such as scales formed during heat treating operations is particularly difficult and requires more work. Additional effort is required to determine the effects of alternate scale removal treatments on metal surface attack pitting and intergranular attack, and on the joining processes.

To circumvent the problem of scale removal, it is proposed that investigations be made of TD NiCr coatings which could be applied before thermal treatment to prevent the formation of characteristic scales. Such coatings should be readily removed from the TD NiCr after thermal processing.

Metallography

The structure of TD NiCr sheet consist of elongated grains. Grain diameters range from 0.01 to 0.200 millimeter.

Conventional metallographic polishing methods may be used. A suggested mechanical procedure would be (1) through 600 grit paper, (2) 6-micron diamond paste on nylon cloth, (3) 6-micron diamond paste on "Duracloth," and (4) a final wheel or vibratory polish with gamma alumina slurry on "Gamal" cloth. For electropolishing, following the nylon cloth step outlined above, a solution of one part sulfuric acid and ten parts methanol at 20 volts would be recommended for sample preparation.

To delineate grain boundaries, electrolytic etching can be employed with a 10-percent oxalic acid solution in water and a potential of 4 volts applied for 3 to 6 seconds.

The metallography of TD NiCr has presented one major problem: the unavailability of any etchant or etching technique which will provide reproducible results. A great deal of effort has been expended to overcome this problem, and such work should be continued. Also, the effect of etching treatments on specimen pitting and grain boundary attack should be determined.

JOINING

General

Thoria-dispersed NiCr has been successfully joined by brazing, spot welding, diffusion bonding, and mechanical fastening. Fusion welding processes are generally not satisfactory for this material, since they cause thoria agglomeration and loss of properties. The preferred joining method is brazing, either furnace or torch brazing, with modified Hastelloy alloys used as filler material. The other preferred processes are diffusion bonding and mechanical fastening.

TIG welding parameters for TD NiCr are being developed. Experimental welds have been made using superalloys as filler metals (e.g., Hastelloy X and IN-718). Fusion causes thoria agglomeration; therefore, simple butt-welded joint efficiency is estimated to be 50 to 70 percent of parent metal strength.

Brazing

1. General. Brazing rather than welding is recommended where possible, since it avoids thoria agglomeration with minimum parent metal interaction. Excellent brazeability has been obtained with high melting brazes such as:

	Braze Temperature, °K (°F)
TD-5, Hastelloy X + 4% Si	1565 (2375)
TD-6, Hastelloy C + 4% Si	1565 (2375)
TD-20, Ni-16%Cr-25%Mo-5%W-4% Si	1565 (2375)

Preliminary tests to determine the remelt temperature of TD-6 braze alloy have been performed. The average remelt temperature was 1640°K (2465°F), with a ±74°K (±25°F) probable range of accuracy. Continued testing is necessary to determine the effect, under load, of repeated post-braze thermal cycling to 1477°K (2200°F) on the remelt temperature, on the base metal structure, and on the braze joint.

Work to date indicates that furnace brazing with TD-6 braze alloy and TIG brazing with Hastelloy X are the most promising. These processes and associated techniques should be further examined to determine the limits of such factors as joint clearance, wetting, flow and erosion characteristics, optimum brazing temperatures, heating rates, and remelt temperatures. Processes should be examined for process reliability and repeatability.

Brazing of TD NiCr to titanium should be investigated to permit attaching TD NiCr heat shield to titanium load structure. Exploratory work has been done to braze tube-to-end fitting joints, and process feasibility has been established. Work should proceed to determine repeatability and reliability for joining these material combinations [11].

2. Braze Material. An extensive brazing alloy investigation has been conducted by the General Electric Company under Air Force Contracts AF33 (615)-1403 and AF33(615)-3476. Braze alloy TD-6 was judged to be the best for TD NiCr material based on mechanical tests, erosion, diffusion, and oxidation studies. It had the highest as-brazed strength and highest strength retention after high-temperature exposure. Because of this extensive development, TD-6 braze alloy was selected for major application studies in this program. This braze material is Hastelloy C with silicon addition. The composition is 17 Mo, 16 Cr, 5W, 4 Si, 1 Al, and the balance Ni, and is in powder form.

A limited application study was made with TD-55, another developmental braze alloy for TD NiCr material. It is Hastelloy X with silicon addition. The composition is 22 Cr, 22 Fe, 9 Mo, 6 Si, 1.5 Co, and the balance Ni, and is in powder form. Nominal brazing temperature is 1530°K (2275°F).

Nominal melting temperature of TD-6 braze alloy is 1570°K (2375°F). Braze remelt temperature was examined in joints with TD NiCr material and established at 1630°K (2475°F). The increase is significant since brazed joints were shown to have an operating temperature higher than indications based on the initial brazing temperature (Table 10) [8].

3. Brazing Procedure. Brazing procedure generally followed the steps itemized below:

a. Preclean TD NiCr test specimen.

b. Assemble test specimen with clips, wire, or tack welds. The tack welds are located at edges where there is little or no stress in the part.

TABLE 10. REMELT TEMPERATURE OF TD NiCr LAP JOINTS BRAZED WITH TD-6 BRAZE ALLOY

Specimen	No. of Brazed Fillets	Remelt Temperature, °K (°F)	Comments
1	2	1613 (2444)	Exceeded solidus, thermocouple failure.
2	2	1634 (2482)	
3	2	>1617 (>2450)	
5	1	1648 (2510)	Thermocouple failure, test aborted.
6	2	>1563 (>2356)	
7	1	1620 (2461)	
Average (of No. 1, 2, 5, 7)		1663 (2474)	

c. Preplace TD-6 braze cement slurry with eyedropper or brush. Apply braze stop-off where required.

d. Place in furnace and perform required thermal cycle.

Compatibility tests were performed on TD NiCr alloy with a variety of hearth materials in order to resolve a problem of TD NiCr diffusion with standard refractory-metal vacuum furnace hearths. Effectiveness of stop-off materials was also examined during compatibility and braze tests. In addition, some comparison brazes were made with TD-55, another developmental braze alloy for TD NiCr alloy. A typical brazed structure is shown in Figure 11 [8].

The generalized technique for brazing with a powdered brazing alloy such as TD-6 is very similar to any furnace brazing procedure. The fundamental difference is that the braze alloy is in powder form and therefore must be cemented in place on an assembly. A most effective method employs an acrylic cement or binder. During the heating cycle this cement decomposes and disappears, leaving no residue; the brazing alloy remains in its prepositioned place. During this program Nicrobraz Cement, a commercial acrylic binder procured from the Wall Colmony Corporation of Detroit, was used exclusively. Application of the powder-cement combination can be made by various methods, including slurry-brush application, slurry-eyedropper method, and spray.

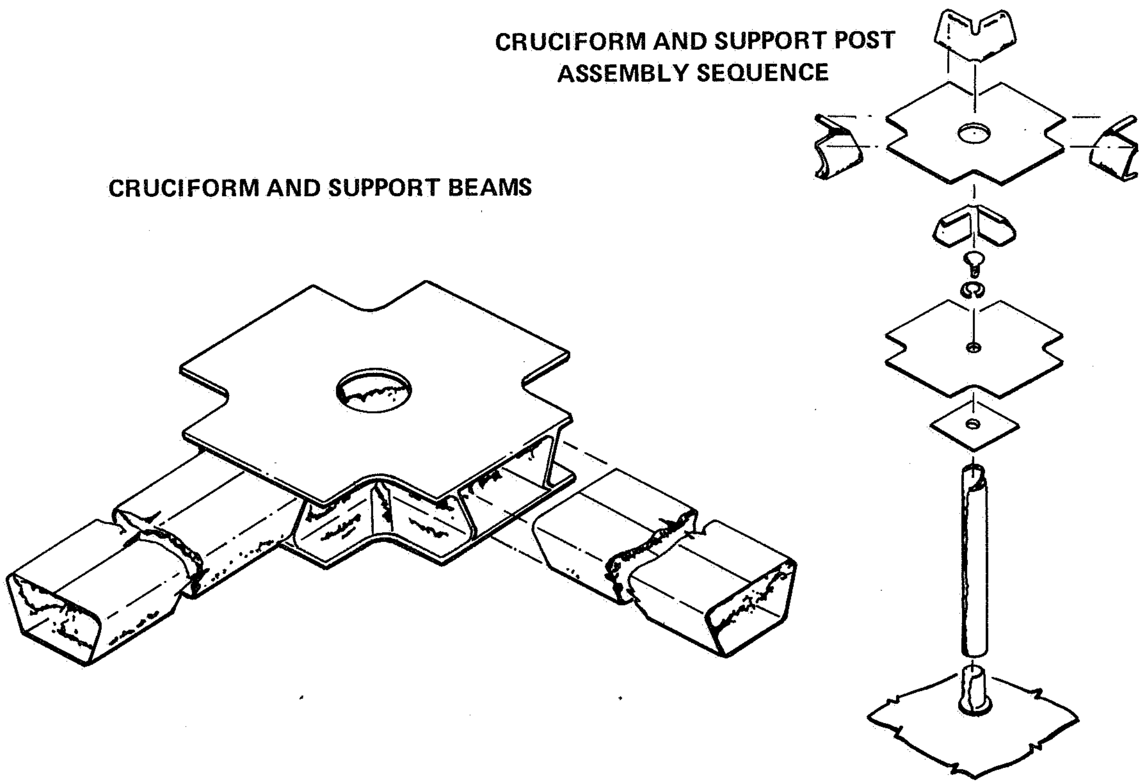

Figure 11. Brazed TD NiCr support structure assemblies for environmental testing of TD NiCr heat shields.

Diffusion Bonding

Diffusion bonding of TD NiCr has been successfully performed, but not without an attendant distortion problem. Creep diffusion bonding offers the opportunity to perform extensive area bonding while minimizing distortion. Creep diffusion bonding should, therefore, be further explored, and its application and limitations for joining TD NiCr structures with minimum distortion be defined. Both diffusion bonding processes (creep and yield controlled) should be fully investigated and qualified as a TD NiCr joining process.

In an attempt to improve the elevated temperature strength of spot-welded TD NiCr sheet, a diffusion spot-bonded process has been developed. This is a two-step process to (1) "stick" weld or forge bond the material using welding electrodes, without a fused nugget, and (2) diffusion bond the stick weld at 1589°K (2400°F) for 5.76×10^4 seconds (16 hr). Results show that diffusion spot-bonded, lap-shear specimens tested at 1477°K (2200°F) for a short-time duration were 175 percent stronger than specimens joined by conventional resistance welds. Work on the improvement of joining processes such as the diffusion spot-bonding process should be continued.

Diffusion Spot Bonding

1. General. Diffusion spot bonding of TD NiCr material has been successfully accomplished by a unique joining method. The first step in the process, after cleaning, is preparation of a stick weld, a weld without a fused nugget but with some joint strength from forge bonding by the welding electrodes. A second step completes diffusion bonding of the stick weld by a thermal treatment, 5.76×10^4 seconds (16 hr) at 1589°K (2400°F), in a helium atmosphere. The joint strength was 175 percent more than the joint strength in a conventional resistance spot weld.

2. TD NiCr Diffusion Spot Bonding. In an attempt to improve the elevated-temperature strength of spot welded TD NiCr, a spot diffusion bonding process has been developed. The first step in producing a diffusion spot bond is to prepare a stick weld. This is a weld without a fused nugget but with some joint strength from forge bonding by the welding electrodes. The weld schedule described in Table 11 [8] was used to stick weld 0.838-millimeter (0.033-in.) TD NiCr with the 200 kVa Sciaky machine.

TABLE 11. DIFFUSION SPOT BOND STICK WELD SCHEDULE

Electrodes 3-A	PG1 at 58
Diameter 15.8 mm (5/8 in.)	PG2 at 18
Tip radius 304.8 mm (12 in.)	PG3 at 18
Weld heat 37	PG4 at 46
Heat cycles 10	ECG1 at 40
Cool cycles 1.5	ECG2 at 0
Weld impulses 12	Motor rotation -5
Electrode force 1361 kg (3000 lb)	Horizontal, spot, both heads
Throat depth 1524 mm (60 in.)	Constant pressure
Arm spacing 508 mm (20 in.)	Constant high
Squeeze timer 0.75	Multiple impulse on
Hold timer 0.75	Intermittent drive on

The second step diffusion bonded the stick weld. The intimate metal contact at the stick weld is a favorable condition for diffusion. The most successful heat treatment used was 5.76×10^4 seconds (16 hr) at 1589 ± 14°K ($2400 {}^{+0}_{-25}$ °F). Atmosphere is important. High vacuum resulted in chromium vaporization. An inert gas atmosphere was used and best results have been with a helium-filled retort.

All indications of the stick weld were removed by the diffusion process. There is a conversion to an interface layer of recrystallized grains. The bond-line indications are removed. The grains are equiaxed and cross the prior bond-line along its entire length.

3. Comparison With Other Joining Methods. The critical applications of TD NiCr are at elevated temperature in the 1256° to 1477°K (1800° to 2200° F) range. Diffusion spot strengths at this temperature have been compared in Table 12 [12] with other joining methods such as conventional resistance spot welds and brazed joints. The comparison is approximate and was made by extrapolating lap-shear data to 8.38-millimeters (0.33 in.) sheet thickness. Diffusion spot bonding is shown to be the most promising method of joining, followed in order by brazing and conventional resistance spot welding. These data are from short-time tests. Long-time tests at elevated temperature are needed for design parameters since diffusion spot bonding showed 175 percent more strength at 1477°K (2200° F) than conventional resistance spot welding.

Mechanical Fasteners [13]

The use of TD NiCr mechanical fasteners in TD NiCr structures will be required for many applications. Preliminary development of TD NiCr rivets for 0.508-millimeter (0.020-in.) thick TD NiCr structure has been conducted and elevated temperature tests were performed. Recrystallization of the manufactured head was noted, although specimens fracture across the base metal rather than by failure of the rivet. Threaded fasteners of TD NiCr, made to Convair designs, have been incorporated into test hardware which was successfully tested through 50 thermal, acoustic, and vibration cycles.

TABLE 12. COMPARISON OF TD NiCr 1477°K (2200°F) LAP-SHEAR STRENGTHS WITH VARIOUS JOINING METHODS FOR 0.838-mm (0.033-in.) THICK SHEET

Data Source	Joining Method	Joint Description	Failure Load per 25.4 mm of Joint, kg (lb)	Stress in Base Metal, N/m^2 (psi)
Convair	Conventional resistance spot welds	Single row spots, 4.57-mm (0.18-in.) dia with 9.14-mm (0.36-in.) spacing	90.7 (200)	4.14×10^7 (6000)
Convair	Diffusion spot bonds (stick weld + heat treat)	Single row spots, 5.842-mm (0.23-in.) dia with 11.68-mm (0.46-in.) spacing	249.5 (550)	1.14×10^8 (16 500)
General Electric AFML-TR67-224	Diffusion spot bond [average of 0.635- and 1.016-mm (0.025- and 0.040-in.) sheet]	Single row spots, 6.858-mm (0.27-in.) dia with 13.72-mm (0.54-in.) spacing	158.7 (350)	7.58×10^7 (11 000)
General Electric AFML-TR67-224	TD-6 brazed joint	3.048-mm (0.120-in.) overlap, 0.1270-mm (0.005-in.) gap	181.4 (400)	8.27×10^7 (12 000)
Solar AFML-TR68-213	TD-6 brazed joint 0.813-mm (0.032-in.) sheet	2.032-mm (0.080-in.) overlap	145.2 (320)	6.76×10^7 (9800)

NOTE: Properties are estimated based on referenced data.

Riveting

The development of a riveting process for the fabrication of the TD NiCr lap joint panel specimen was performed by Martin Marietta Corporation [11]. The results of their studies are summarized in the following paragraphs.

The hole filling capability of 6.35-millimeter (0.250-in.) rivets proved to be adequate with the rivet shank electropolished to 6.25/6.30-millimeter (0.246/0.248-in.) diameter and the hole reamed to 6.325/6.375-millimeter (0.249/0.251-in.) diameter.

Thoria-dispersed NiCr rivet test results appear to be satisfactory when high surface quality electropolished and machined flush head rivets are used. Previous tests at 700°K (800°F) preheat indicated some improvement in compressibility. However, defect-free rivets formed satisfactorily at room-temperature conditions. A squeeze operation loading to 6350.4 kilograms (14 000 lb) on a Tinius-Olsen testing machine produced a good driven-end head configuration. Suitable quality rivets were selected after electropolishing. An improved extruding process is now available whereby the 76.20-millimeter (3-in.) billet can be reduced to 6.35-millimeter (0.25-in.) diameter in one pass. This condition controls the cold work, optimizes grain size, and produces a superior product over the formed method.

In summary, the following observations may be made:

1. Good quality rivets can be squeeze riveted to a minimum strength of 1678.3 kilograms (3700 lb) is surface quality is acceptable.

2. Rivet stock supplied was defective, containing inclusions and cracks.

3. Thoria-dispersed NiCr alloy rivets are readily installed at room temperature.

4. Electropolishing revealed defective surface condition of the rivets.

5. Greater installation force is required for TD NiCr alloy than is normally used for riveting processes.

CONCLUSIONS

Brazing parameters to join TD NiCr material with TD-6 braze alloy are 120 to 240 seconds (2 to 4 min) at 1589° to 1594°K (2400° to 2410°F) and 1.3×10^{-3} N/m^2 (10^{-5} torr). The wetting and flow of these material combinations are rated excellent. Braze alloy TD-6 has excellent ability to wet and flow between wide faying surfaces with near-zero clearances. Gaps of 0.152 millimeter (0.006 in.) were successfully bridged with TD-6 braze alloy.

The remelt temperature of TD-6 braze alloy is 1630.5°K (2475°F), compared with the 1589°K (2400°F) brazing temperature. This increase is significant because the application temperature of TD NiCr brazed assemblies is increased.

Diffusion spot bonding of TD NiCr material is a very promising method of forming. At 1477°K (2200°F) the shear strength is 2.75 times that of a conventional resistance spot welded joint.

TD NiCr is a promising material for heat shield applications with a maximum temperature of 1477°K (2200°F). Under normal stress/exposure cycling the material in both the longitudinal and transverse grain directions has a useful life well in excess of 25 cycles. Under an abort stress condition, failure has been observed with transverse specimens during the third or fourth cycles. This, however, is acceptable because a heat shield panel would not normally be subjected to more than one abort condition before replacement. In material exposed under stress for 25 cycles, no internal oxidation was visible in the microstructure.

Female die forming of TD NiCr material is required initially, before male die forming can be successfully accomplished.

Thoria-dispersed NiCr beaded heat shield panel material, 0.305 millimeter (0.012 in.) thick, requires a pressure plate on the panel border for control of panel buckling from forming operations. Edges of the pressure plate must be as close as possible to the bead configurations to forestall buckling.

Use of a caul sheet is a key factor in successful forming of 0.305-millimeter (0.012-in.)-thick TD NiCr beaded panels. The caul sheet at all times must support the workpiece in male and female die forming operations.

Slotting the beaded panel border aided flattening by planishing operations after forming.

Thoria-dispersed NiCr material in the as-received condition demonstrates uniform elongation in the range of 10 to 11 percent in 25.4 millimeters (1 in.), which is approximately 50 percent of the ductility measured in standard tensile coupons. This is probably the upper formability limit for material in the as-received condition.

Stress-relieving TD NiCr between tensile strain applications contributes to an increase in formability limits. Material has been stretched a maximum of 13.5 percent elongation in 25.4 millimeters (1 in.) when the material received two stress-relieving treatments. The elongation is approximately 65 percent of the ductility measured in standard tensile coupons.

RECOMMENDATIONS

1. The formability limit of TD NiCr material has not been completely examined. In 1967 some studies were conducted wherein it was shown that TD NiCr could be stretched approximately 10.5 percent in 25.4 millimeters (1 in.) without stress relieving between forming stages. Material in beaded panels has been stretched to a maximum elongation of 13.5 percent in 25.4 millimeters (1 in.) after two stress-relieving operations between forming stages. Continued stretching capability is believed likely after additional stress relieving. A parametric study of strain effects as a function of several stress-relieving treatments between strain applications is recommended. The investigation would show (a) mechanical properties after tensile strain, (b) the relationship of yield and ultimate tensile strengths, knowledge of which is desirable for successful forming, and (c) the uniform percent elongation to establish formability limits. The data would reveal conditions for maximum structural efficiency of TD NiCr material which would be useful for manufacturing technology and engineering design.

2. Remelt temperature of brazed TD NiCr material can be increased approximately 311°K (100°F) above the nominal brazing temperature of the braze alloy. A study is recommended to determine if the remelt temperature is further increased by thermal treatments or other brazing techniques to provide usable brazed joints at the upper temperature limit of TD NiCr material.

3. A unique method has been developed to join TD NiCr material by a diffusion spot bonding process. Strength of the diffusion spot bond at 1477°K (2200°F) was 175 percent more than the strength of a conventional resistance

spot weld. The strength increase is very substantial. Long-time temperature exposures, including cyclic temperature regimes, are recommended. The use of interleaf material at the faying surface is also recommended in the exploration of this interesting joining method.

4. The beaded heat shield panels can be formed in conventional hydraulic press equipment. Forming rates are relatively slow in comparison with high-energy forming equipment such as the electrohydraulic process. It is recommended that TD NiCr beaded heat shield panel forming using this equipment be evaluated to verify maximum formability limits and cost effectiveness of the forming system. Since the ductility of TD NiCr material is unaffected by a wide range of strain rates, it should be amenable to high-energy forming processes.

5. Additional areas requiring investigation include: (a) the effect of strain rate on TD NiCr formability and (b) the feasibility of using high-energy rate forming to achieve improved forming depths and detailing, or to minimize distortion. In both cases, preliminary investigations have indicated that further work should prove rewarding.

6. A final forming investigation needs to be initiated to develop techniques for economically producing simple and compound curved TD NiCr heat shield panels. One approach would be to fabricate flat panels and to hot creep form them to the desired contour. The conventional approach would be to form all details to the desired configurations, and then to join them.

7. An in-depth study of additional stress relieving treatments after varying strain applications should be conducted to determine the magnitude of maximum formability from increased ductility.

8. The effect of various amounts of cold working (prestrain) on the mechanical properties of TD NiCr sheet supplied in the stress-relieved [1561°K (2350°F)] condition has also been determined in preliminary tests. However, continued work should be performed to determine the prestrain limits and the combined effects of interstage heat treatments and subsequent cold work on the mechanical properties, particularly for thin-gage sheet.

9. Tests of processes should be performed to develop meaningful and useful design data. Static tensile tests for TD NiCr base metal and static lap and cross tension tests for brazed diffusion bonded, spot-welded, and mechanically fastened joints should be performed. These tests should be performed at room and elevated temperatures, both with and without exposure

to a simulated reentry temperature-stress-pressure profile. Figure 12 illustrates such a simulated profile. Elevated temperature creep tests at various temperatures and stress levels, up to and including the maximum use temperature, should also be performed.

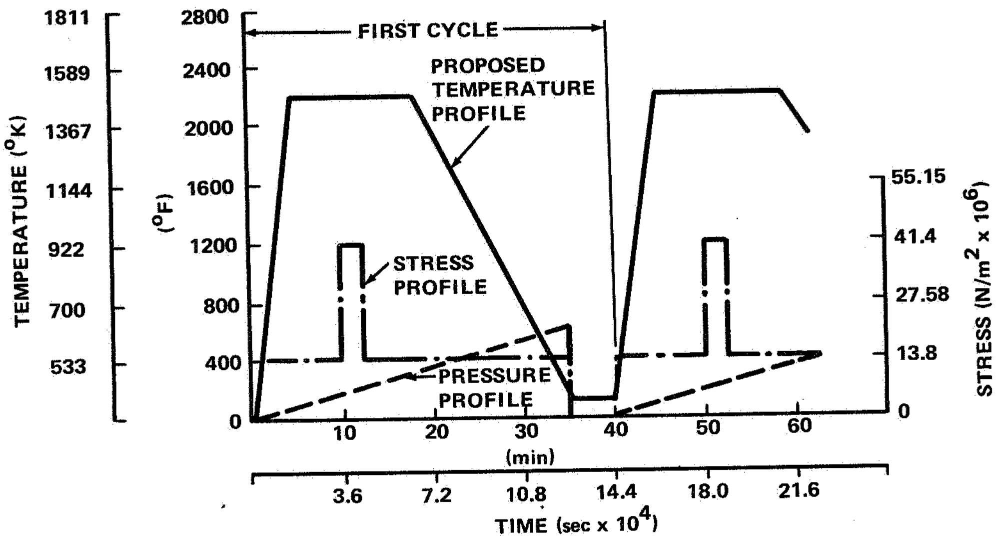

Figure 12. Simulated reentry testing profiles.

APPENDIX

MATERIALS SPECIFICATION OF THORIATED NICKEL BASE ALLOYS; GENERAL – ALL ALLOYS – SHEET

1. Scope

1.1 This specification covers surface and dimensional requirements for all Fansteel thoriated nickel alloys in sheet form.

2. Quality

2.1 Micro or metallographic examination shall reveal a substantially uniform structure. Material shall be essentially free of porosity. Standards for acceptance or rejection shall be as agreed upon by purchaser and vendor.

3. Structural Condition

3.1 Material will normally be supplied in the stress-relieved condition.

4. Surface Finish

4.1 Material may be supplied with a cold-rolled (and heat-treated), pickled, or polished surface.

4.2 Surface defects may be removed by such methods as grinding, buffing, or polishing.

4.3 By visual inspection, the material shall be free of any evidence of contamination. Differences in reflectivity shall not be considered evidence of contamination.

4.4 No pits which reduce the thickness of the material below the minimum thickness specification are acceptable. Occasional pits of less than this depth are acceptable provided the number is not in excess of fifty per square meter (five per square foot). Individual pits or roughened areas which appear under magnification as a scattering of pits shall be acceptable if not of measurable depth mechanically [i.e., are less than 0.012 millimeter (0.0005 in.) in depth as determined optically by the light section method]. There is no restriction on the number of pits of this type which may be present.

4.5 No mars, gouges, scratches, or similar defects which reduce the thickness of the material below the minimum thickness specification are acceptable. Occasional defects of this type less than this depth are acceptable provided the number is not in excess of 50 per square meter (5 per square foot). Superficial scratches of a depth not measurable mechanically [i.e., less than 0.012 millimeter (0.0005 in.) in depth as determined optically by the light section method] shall not be cause for rejection in any number.

5. Dimensional Tolerances

5.1 Thickness Tolerances:

Thickness (mm)	Thickness (in.)	Thickness Tolerance (mm)	Thickness Tolerance (in.)
0.0152-0.254	0.006-0.010, incl.	0.0375	±0.0015
Over 0.254-0.635	Over 0.010-0.025, incl.	0.051	±0.002
Over 0.635-0.864	Over 0.025-0.034, incl.	0.076	±0.003
Over 0.864-1.422	Over 0.034-0.056, incl.	0.102	±0.004
Over 1.422-1.778	Over 0.056-0.070, incl.	0.127	±0.005
Over 1.798-1.981	Over 0.070-0.078, incl.	0.152	±0.006
Over 1.981-2.769	Over 0.078-0.109, incl.	0.178	±0.007
Over 2.769-3.560	Over 0.109-0.140, incl.	0.203	±0.008
Over 3.560-4.345	Over 0.140-0.171, incl.	0.229	±0.009
Over 4.345-4.748	Over 0.171-0.187, incl.	0.254	±0.010

In accordance with standard commercial practice, thickness measurements are taken at least 9.53 millimeters (3/8 in.) from the edge of the sheet.

5.2 Width Tolerance:

Width	Width Tolerance
Up to 711.2 mm (28-in.) incl.	+3.175 mm - 0 (+ 1/8 in., - 0)

5.3 Length Tolerance:

Length	Length Tolerance
Up to 2438.4 mm (96 in.) incl.	+3.175 mm - 0 (+ 1/8 in., - 0)

5.4 Flatness Tolerance: The maximum deviation from flatness shall not exceed 8 percent as calculated from the following formula:

$$\frac{H}{L} \times 100 = \text{percent flatness deviation,}$$

where

H = maximum vertical distance between a flat reference surface and the lower surface of the sheet

L = minimum horizontal distance between the high point on the sheet where H is determined and the point where the lower surface of the sheet touches the reference.

A value of H less than 1.59 mm (1/16 in.) shall not be cause for rejection.

The maximum deviation from flatness shall not exceed 50.8 mm (2 in.), independent of span, as measured with the sheet resting concave side down on a flat reference surface.

6. Marking

6.1 Unless otherwise specified, each sheet shall be marked with Fansteel alloy identification, compaction number, and nominal thickness in millimeters (inches). The characters shall be at least 9.53 millimeters (3/8 in.) in height and applied with a marking fluid removable by cleaning solution. The rows shall be alternately staggered and spaced not more than 76.2 millimeters (3 in.) apart. Direction of final rolling shall be marked.

7. Packaging

7.1 Sheet shall be protected by interleaving with paper between each sheet and the next. Material is normally packaged in wooden boxes reinforced with steel strapping. Small orders may be packaged with corrugated paper stiffeners and Kraft paper wrapping.

7.2 Markings on the outside of the package shall include customer's name and purchase order number and Fansteel thoriated nickel alloy designation.

8. Reports

8.1 Each shipment shall be accompanied by a shipping memorandum which reports the customer purchase order number, alloy identification number, and size of pieces, heat number, and weight.

8.2 Each shipment shall be accompanied by three copies of a test report giving the customer purchase order number, alloy identification, specification number, compaction number, chemical composition, structural condition, and mechanical properties.

9. Rejection

9.1 Fansteel must be notified in writing of all rejection claims within 90 days of receipt of material. This notice should contain the purchase order number, compaction number, size, weight, number of pieces rejected, and detailed reasons for rejecting claims. Fansteel shall have the opportunity of inspecting this material at the customer's plant to determine the validity of the rejection claim. Upon authorization by Fansteel, the rejection material shall be returned as directed at the expense of Fansteel.

10. Shipping Tolerance

10.1 A shipping weight tolerance of ± 10 percent of ordered weight shall apply.

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A STATE-OF-THE-ART LITERATURE SURVEY OF THORIA-DISPERSED NiCr FOR THE SPACE SHUTTLE THERMAL PROTECTION SYSTEMS

By W. L. Hayes

The information in this report has been reviewed for security classification. Review of any information concerning Department of Defense or Atomic Energy Commission programs has been made by the MSFC Security Classification Officer. This report, in its entirety, has been determined to be unclassified.

This document has also been reviewed and approved for technical accuracy.

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